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PATENT SPECIFICATION

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COMPLETE SPECIFICATION

Detergent Tablets

We, COLGATE-PALMOLIVE COMPANY, a Corporation organised and existing under the Laws of the State of Delaware, United States of America, of 300 Park Avenue, New York 22, New York, United States of America, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the coating of detergent tablets. More particularly, this invention relates to a process by which a coloured coating is uniformly applied to a detergent tablet. The invention also relates to detergent tablets covered with a homogeneous coloured coating.

According to one aspect of the present invention, a process for producing a coloured coating of water-soluble polymer on a detergent tablet of adherent particulate composition of water-soluble organic detergent and water-soluble inorganic salt comprises applying to a surface of the detergent tablet a mixture of a water-soluble film-forming organic polymer, a colouring material which is soluble or dispersible in water and an organic surface active compound in an aqueous liquid medium and drying the surface of the tablet to form thereon a coloured solid coating of substantially uniform appearance.

According to another aspect of the invention a detergent tablet of uniformly coloured appearance comprising a body of adherent particulate composition of water-soluble synthetic organic detergent and water-soluble inorganic salt, and a homogeneous solid coating of water-soluble organic polymer, a water-soluble or water-dispersible colouring material and an organic surface active compound.

It has been found that solutions of water-soluble organic film-forming polymers, such as polyvinyl alcohol, when they contain colouring materials, often do not, by themselves, consistently produce a uniformly coloured coating on detergent tablets. Instead, a speckling effect often results which is considered undesirable. However, when a polymer-containing coating composition which also contains a synthetic organic surface active compound in accordance with the invention is applied, a uniformly coloured coating of the tablet is obtained. This beneficial result is unexpected, in view of the fact that the detergent tablet itself, to which the coating is applied, includes a substantial proportion of organic surface active compound.

Detergent tablets which may be coloured by the invented process are those which include water-soluble organic detergent and water-soluble inorganic salt. The organic detergent may be either an anionic detergent or a nonionic detergent. Among the suitable anionic detergents are the higher alkyl benzene sulphonates, higher fatty alcohol sulphates, higher fatty acid monoglyceride sulphates, alpha sulphonated higher fatty acids, higher fatty acid amides of N-methyl taurine, higher fatty acid soaps and sulphated polyethoxy detergents. Among the nonionic detergents may be mentioned the ethoxylated alkyl phenols (Igepals), block copolymers of ethylene oxide and propylene oxide (Pluronics), reaction products of higher fatty alcohols and lower alkylene oxides (Emulphogene), polyoxyethylated higher fatty acids (Emulphor, trade mark), and polyethoxy and poly lower alkoxy esters and ethers of sugar alcohols, especially of sorbitol and mannitol. Among the preferred anionic detergents is sodium tridecyl benzene sulphonate, a mixture of alkyl benzene sulphonates in which the alkyl component is derived from a mixture of propylene tetramer and pentamer. The higher fatty acid soap

which may be employed is usually the sodium soap of a mixture of tallow and coconut oil fatty acids. A preferred nonionic detergent is an alkyl phenoxy poly lower alkanol, such as nonyl phenoxy polyoxyethylene ethanol of 9 to 10 ethoxy groups per molecule. This compound is a preferred one of such useful nonionic detergents which have an alkyl group of 6 to 14 carbon atoms and an ethoxy chain 4 to 12 units long, each unit comprising from 2 to 4 carbon atoms.

The anionic detergents are usually employed in the form of a water-soluble salt, preferably an alkali metal salt, such as a sodium or potassium salt. Of course, in making a solid product it is desirable for the detergents used to be solid or to be such that they will allow the production of a satisfactory solid product.

In the body of the detergent tablet is included a water-soluble inorganic salt. The water-soluble salt aids in the production of a satisfactory, strong tablet and often acts as a builder to assist the organic detergent in its cleaning action. Among the inorganic salts which may be used are pentasodium tripolyphosphate, tetrasodium pyrophosphate, sodium phosphate and corresponding acid salts of phosphoric acid, sodium silicate, e.g., one with (Na_2O/SiO_2 ratio of 1:2.35), sodium carbonate, sodium sulphate and sodium chloride. Other water-soluble alkali metal salts, such as the potassium salts, may be employed in suitable circumstances.

The body of the detergent tablet is preferably made by spray drying an aqueous crutcher mixture of synthetic detergent and inorganic salt to solid beads, which are usually within the size range of 8 to 100 mesh, United States Standard Sieve Series, but particles of detergent and particles of salts may be compacted by other suitable means to form the tablet bodies. The particles may be treated before lightly compacting, preferably by spraying them with water, a solution of sodium silicate or other suitable treating agent and sometimes by additionally incorporating talc in the mix. Compacting is preferably done at light pressures, under 100 pounds per square inch, preferably 40 to 90 pounds per square inch.

The proportions of detergent and inorganic salt in the tablets are regulated to obtain a satisfactory and economical product. Usually, the proportion of detergent in the body of the tablet will be from 10 to 40% thereof and the proportion of inorganic salt may be from 30 to 90%, any balance of the composition being other suitable adjuvants, including perfume, moisture, fluoroescent brighteners, foaming agents, bactericides, sequestrants and so forth.

The polymer, colouring, surface active material and liquid medium to be applied to a surface of the tablet described above are preferably in the form of a liquid which is of sufficient fluidity to be sprayed. Preferably, the tablets are moved on a mesh belt underneath spray nozzles which are set so as to spray onto a face and a portion of the sides of the tablets as they are moved under the nozzles. Spraying is done at high pressure, e.g., 1,000-1,200 pounds per square inch at the spray nozzle tip, and the sprays employed are usually of the full fan type. The solution or dispersion being sprayed is desirably at a temperature between 10°C. and 90°C., usually of 40°C. to 85°C. and preferably of 60°C. to 80°C. The tablets may be at room temperature or warmer, usually up to 50C. Under normal circumstances, it is not necessary to heat the tablets. It has been found that a high pressure spray unit creates a satisfactory spray of liquid coating composition at the above conditions and produces a smooth film on the detergent tablets. The particle sizes of the sprayed material may cover a range but the major part by weight of these is usually considered to be in the range 100 to 1,000 microns in diameter, many of the particles being so light that they settle only slowly in air. After spraying of a face and sides, the tablet is inverted and the uncoated areas are then treated. It is highly preferred to have the entire tablet covered by the coating composition but, if desired, only a portion thereof may be coated.

The composition which is sprayed onto the detergent tablet includes a water-soluble organic polymer, preferably a film-forming synthetic organic polymer which will dissolve readily in water and therefore not detrimentally impede the disintegration of the detergent tablet in the wash water. As examples of these materials may be named polyvinyl alcohol, ethoxylated polyvinyl alcohol, sodium carboxymethyl cellulose, hydroxypropyl methyl cellulose, hydroxyethyl cellulose and polyvinylpyrrolidone. Of these, the most preferred is polyvinyl alcohol, which produces a smooth readily soluble film coating on the detergent tablet. The polyvinyl alcohol employed may include a minor percentage, e.g., 15 to 30%, of polyvinyl acetate.

The colouring material may be a suitable dye or pigment. If a pigment is employed, it should be water-dispersible. Such pigments are very finely divided materials, usually of micron sizes and usually have present, mixed intimately with them, a wetting agent, so that they will not form lumps of coloured material. Among the suitable pigments, one which is highly preferred is a phthalocyanine pigment which is usually employed as a copper or iron compound, and is highly stable. Such a pigment is sold under the trade names Heliogen Blue WD or Monastral Blue. Of course, other pigments of satisfactory characteristics may also be used. So far as the dyes are concerned, one may use any which are sufficiently light-stable and alkali-stable to maintain their colour satisfactorily when applied to the detergent

tablets. Erio cyanine blue dye (Geigy) produces a satisfactory coloured coating but should preferably be employed on neutral tablets. Of course, the dyes should be water-soluble and should not objectionably colour materials which are washed with the detergent tablets.

The organic surface active compound of the coating composition is either an anionic or nonionic material, the cationics tending to react with dyes, detergents and materials with which the product comes into contact more than do the non-cationic compounds. Although the wetting agents, which include compounds having shorter alkyl or acyl chains on a hydrophilic structure, are surface active materials and come within the scope of this invention, it is preferred to employ those surface active compounds which are water-soluble and possess a higher fatty alkyl chain, i.e., an alkyl group of 8 to 18 carbon atoms. Among these are the higher alkyl benzene sulphonates, of which the most preferred is sodium tridecyl benzene sulphonate, the tridecyl benzene sulphonate being a mixture of benzene polymers of 12 to 15 carbon atoms. Instead of the higher alkyl benzene sulphonates one may also use the dioctyl sulphosuccinates, the polyethoxy ethanol derivatives of nonyl phenol (Igepal), water-soluble soap (85% tallow, 15% coconut oil soap), monoglyceride sulphate, or other detergents. It is preferred to use an anionic detergent which is a sulphated or sulphonated compound and of these the water-soluble alkyl benzene sulphonates have been found to be generally superior. As was previously mentioned, the detergents are employed in a water-soluble form and the anionic detergents usually may be solubilized by being converted to their alkali metal salts, e.g., sodium salts.

The liquid medium for the polymer, colouring material and surface active agent should be such as to produce a liquid composition. Preferably the liquid medium is water, although other materials may be employed with water to regulate the viscosity and other characteristics of the composition. It has been found desirable to use small proportions of glycerol, for its plasticizing effect. Usually this material should be present in concentrations of between 1 and 10% in the coating composition.

The proportions of the various constituents in the coating composition should be kept within limits to obtain a product which will form a satisfactory coating and which can be readily applied to the tablets by practicable methods. Thus, the proportion of film-forming polymer in the coating solution or dispersion should be between 5 and 30% thereof, the surface active compound should be 0.15 to 10% and preferably 1 to 10% of the solution, and the colouring material should be present in sufficient proportion noticeably to colour the coating on the detergent tablet, i.e., the proportion of colouring material should be between 0.001 and 5%. The lower limit recognizes the very high colouring power of some dyes. Usually the lower limit will be 0.01% while if pigments are used the least proportion employed will usually be about 0.1% The proportion of surface active agent will be enough to promote continuous covering of the surface of the detergent tablet by the coating composition and to prevent coalescing of the coating after application. If such coalescing took place, the detergent tablets would appear speckled rather than of uniform continuous colouring, due to a thinning out of the coating in spots. In the concentration specified, the coating liquid will form a substantially uniform, thin film through which the colouring material is evenly distributed and which film is evenly distributed over the surface of the detergent tablet. The liquid medium is usually present in a proportion of from 40 to 94% of the coating liquid, and preferably of 50 to 94% thereof.

Within the broader ranges of constituents mentioned, it will be realized that preferred ranges result in a better product. Thus, if the percentage of polyvinyl alcohol is kept at about 15 to 20% (including polyvinyl acetate), colouring material is at about 0.1 to 1%, alkyl benzene sulphonate is at 1 to 3% and the balance of the composition is water with a small proportion of glycerol and inorganic salt, an excellent coating composition results.

The coating is applied to the detergent tablets to form a thin coloured film on the surfaces of the tablets. This film will normally comprise about 0.2 to 3% of the weight of the dried coated tablets and will result from the application of 0.4 to 10%, and preferably 1.5 to 5%, by weight of the coating liquid.

After the coating composition has been applied evenly to the surface of the detergent tablet, the tablet is dried, for instance by infra-red heating lamps, with air circulated over the tablet surfaces. It is dried while the coating composition is still uniformly distributed over the surfaces of the tablet, causing the production of a uniformly covered homogeneous coating, usually between 0.01 and 0.8 millimetres thick, without coalescing or speckling. During the drying operation, the temperature of the tablet surface may be elevated to as high as 90°C. or evaporation may be caused at a lower temperature. The products, especially those made with water-dispersible pigment, have a uniform coloured coating which is of excellent stability and does not fade on storage.

The following example illustrates the invention. Percentages given in the example and elsewhere in the specification are by weight.

EXAMPLE

	%
Nonyl phenol ethoxylate (9.5 ethylene oxide units to the molecule)	13
Sodium tridecyl benzene sulphonate	5
Technical cetyl alcohol	1
Sodium tripolyphosphate	43
Sodium sulphate	25
Sodium silicate (Na_2O/SiO_2 ratio of 1: 2.35)	4
Moisture	8.5
Adjuvants (anti-redeposition agent, fluoroescent brightener, perfume, anti-oxidant)	0.5
	100.0

The above formula was made by spray drying an aqueous crutcher mix slurry of all materials except perfume, which was added after spraying. The particle sizes produced were substantially within the range of 8 to 100 mesh, United States Standard Sieve Series. In a tumbling drum there were added an aqueous sodium silicate solution, (43.5% of silicate solids of 1:2.35 Na_2O/SiO_2 ratio) and talc powder, to result in a coating of the detergent beads with 7.5% of the silicate and 2% talc.

The detergent beads were then tabletted to flat cylinder form by pressing between forming members at a pressure between 40 and 90 pounds per square inch, following which they were sprayed with a coating of the following composition:

	%
Polyvinyl alcohol (containing 15—30% polyvinyl acetate)	17.0
Phthalocyanine blue pigment powder, water dispersible	0.2
Sodium tridecyl benzene sulphonate (tridecyl denoting a mixture of propylene tetramer and pentamer)	1.4
Sodium sulphate	0.2
Glycerol	2.5
Water	78.7
	100.0

The coating composition was made by mixing with the other components an aqueous solution of the surface active agent, containing the sodium sulphate. Such addition, as a solution, minimized objectionable foaming during mixing.

The coating solution, heated to 74°C., was sprayed onto a face and two sides of a detergent tablet through a spray nozzle at about 1,000 pounds per square inch nozzle pressure. The spray was of full fan shape. The detergent tablets were passed at room temperature.,

20°C., under the sprays on a moving belt and were dried under infra-red lamps. Both faces and all sides were coated in this manner. The drying took place immediately after coating, so the coating was substantially uniform in thickness, about 0.2 millimeters thick. Neither the coating nor the colour had coalesced at the time of drying.

The product made was smooth surfaces and of a uniform blue appearance. It was form-retaining and strong enough to withstand shipping. It was readily disintegrable in wash water and washed clothing well.

In similar experiments, the concentrations of constituents were modified, within the ranges disclosed in this specification, with similar results. When Erio cyanine blue dye was substituted for the phthalocyanine blue pigment, 0.006% of the dye replacing 0.2% pigment, a satisfactory uniformly coloured product was obtained. However, the pigments appear to be more stable during storage. Although acceptable results were obtained when the spray was at 49°C. and 82°C., better conditions resulted at 68°C. and 74°C. Although other spray nozzles may be employed, it is thought that the sprays which deposit droplets of 100 to 1,000 microns diameter are best.

In addition to alkyl benzene sulphonate as the surface active agent, similar experiments were run, with acceptable results, with sodium dioctyl sulphosuccinate and sodium higher fatty acid soap, to name only two of the more successful agents. Nonyl phenol polyethoxy ethanol (Igepal CO-630 of 9-10 ethoxy groups) helps to make a uniform coating but is not superior to alkyl benzene sulphonate. In fact, of all the surface active materials tried, the alkyl benzene sulphonates appear to give best uniform colouring and coating, and are preferred.

WHAT WE CLAIM IS:—

1. A process for producing a coloured coating of water-soluble polymer on a detergent tablet of adherent particulate composition of water-soluble organic detergent and water-soluble inorganic salt, which comprises applying to a surface of the detergent tablet a mixture of a water-soluble film-forming organic polymer, a colouring material which is soluble or dispersible in water and an organic surface active compound in an aqueous liquid medium, and drying the surface of the tablet to form thereon a coloured solid coating of substantially uniform appearance.

2. A process as claimed in Claim 1 in which the mixture is applied to the detergent tablet at a temperature between 10°C and 90°C., the surface active compound being present with the film-forming synthetic organic polymer and colouring material in a proportion which improves the power of the dispersion to cover the surface of the detergent tablet uniformly, and in which the mixture is thereafter heated on the surface of the tablet to dry the mixture while it is evenly coating a substantial proportion of the tablet.

3. A process as claimed in Claim 1 or Claim 2 in which the organic detergent is an anionic or nonionic detergent, and the water-soluble inorganic salt is a phosphate, silicate or carbonate builder salt.

4. A process as claimed in any of the preceding claims in which the polymer, colouring material and surface active compound are sprayed on the detergent tablet as fine droplets of an aqueous solution or dispersion thereof, at a temperature of between 40 and 85°C.

5. A process as claimed in any of the preceding claims in which the surface active compound is an anionic or nonionic synthetic organic detergent compound and comprises 0.15 to 10 per cent by weight of the mixture, the film-forming organic polymer is present in a proportion of from 5 to 30 per cent by weight of the mixture, and the colouring material is present in a sufficient proportion in the mixture noticeably to colour the coating on the detergent tablet.

6. A process as claimed in any of the preceding claims in which the film-forming polymer is selected from polyvinyl alcohol, ethoxylated polyvinyl alcohol, sodium carboxymethyl cellulose, hydroxypropyl methyl cellulose, hydroxyethyl cellulose and polyvinylpyrrolidone, the colouring material is a water-soluble dye or a finely divided water-dispersible pigment, and the surface active compound is a synthetic organic anionic surface active detergent having an alkyl chain of 8 to 18 carbon atoms.

7. A process as claimed in any of the preceding claims in which the detergent tablet is a tablet of compacted particles and the mixture is sprayed onto the tablet and comprises 5 to 30 per cent of the polymer, 0.001 to 5 per cent of colouring material, 1 to 10 per cent of the surface active compound and 40 to 94 per cent aqueous medium, all by weight based on the mixture.

8. A process as claimed in any of the preceding claims in which the surface active compound is a water-soluble alkyl benzene sulphonate in which the alkyl group is of 12 to 15 carbon atoms.

9. A process as claimed in any of the preceding claims in which the mixture applied to the surface of the tablet amounts to 0.4 to 10 per cent by weight, based on the weight of the dried, coated tablet, and comprises by weight based on the mixture, 5 to 30 per cent of polyvinyl alcohol, 0.01 to 5 per cent of finely divided water-soluble dye or water-dispersible pigment, 1 to 10 per cent of a water-soluble alkyl benzene sulphonate in which the alkyl group is of 12 to 15 carbon atoms and 50 to 94 percent water.

10. A process as claimed in any of claims 1 to 8 in which the mixture is an aqueous dispersion and is applied to the surface of

the tablet at a temperature of between 60°C. and 80°C. and comprises by weight based on the mixture, 5 to 30 per cent of polyvinyl alcohol, 0.1 to 5 per cent of finely divided water-dispersible pigment, 1 to 10 per cent of sodium alkyl benzene sulphonate in which the alkyl group is a propylene polymer of 12 to 15 carbon atoms, 0.1 to 10 per cent of an alkali metal sulphate and 50 to 94 per cent water.

11. A process as claimed in any of the preceding claims in which the colouring material is a finely divided water-dispersible phthalocyanine pigment.

12. A process as claimed in any of the preceding claims in which an aqueous liquid is applied to the surface of the tablet and is made by mixing with the other necessary constituents an aqueous solution of neutralized alkyl benzene sulphonic acid.

13. A detergent tablet of uniformly coloured appearance which comprises a body of adherent particulate composition of water-soluble organic detergent and water-soluble inorganic salt, and a homogeneous solid coating of water-soluble organic polymer, a water-soluble or water-dispersible colouring material and an organic surface active compound.

14. A detergent tablet as claimed in claim 13 in which the particulate composition is compacted, the organic detergent is a synthetic non-cationic detergent and the water-soluble inorganic salt is a phosphate, silicate or carbonate builder salt.

15. A detergent tablet as claimed in claim 13 or claim 14 in which the homogeneous coating is 0.2 to 3% by weight of the tablet.

16. A detergent tablet as claimed in any of claims 13 to 15, in which the homogeneous coating comprises 5 to 30 parts by weight of a water-soluble film-forming synthetic organic polymer selected from polyvinyl alcohol, ethoxylated polyvinyl alcohol, sodium carboxymethyl cellulose, hydroxypropylmethyl cellulose, hydroxyethyl cellulose and polyvinylpyrrolidone, 0.001 to 5 parts by weight of a water-soluble dye or a finely divided water-dispersible pigment, and 1 to 10 parts by weight of an organic anionic surface active detergent having an alkyl chain of 8 to 18 carbon atoms.

17. A detergent tablet as claimed in any of claims 13 to 16, in which the tablet is composed of lightly compacted spray dried particles, the colouring material is a finely divided water-dispersible phthalocyanine pigment, the surface active agent is a sodium alkyl benzene sulphonate the alkyl group thereof being a propylene polymer of 12 to 15 carbon atoms, and the homogeneous coating also contains 1 to 10 parts by weight of glycerol.

18. A detergent tablet made by a process as claimed in any of claims 1 to 12.

19. A process for producing a coloured coating of water-soluble polymer on a detergent tablet substantially as described in the foregoing example.

20. A detergent tablet substantially as described in the foregoing example.

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